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Indian Standard

SPECIFICATION FOR DRINKING CHOCOLATE (SWEETENED COCOA POWDER)

(Second Revision)

UDC 663·918·22

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 6 *June* 1987

Indian Standard

SPECIFICATION FOR DRINKING CHOCOLATE (SWEETENED COCOA POWDER)

(Second Revision)

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Indian Standard

SPECIFICATION FOR DRINKING CHOCOLATE (SWEETENED COCOA POWDER)

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 30 August 1986, after the draft finalized by the Stimulant Foods Sectional Committee had been approved by the Agricultural and Food Products Division Council.

0.2 Since the first revision of this standard in 1979, the need for updating the standard in the light of current trade practice and the knowledge obtained in its use, has been felt. In this revised version title of the Indian Standard has been changed to 'Drinking Chocolate' (Sweetened Cocoa Porwder) and microbial limits have also been specified in order to produce drinking chocolate hygienically.

0.3 Drinking chocolate (sweetened cocoa powder) is one of the ready-to-make beverages which are increasingly used in the country and abroad. This is cocoa powder sweetened with carbohydrates.

0.4 In the preparation of this standard assistance has been derived from the internationally accepted methods of analysis. Due consideration has also been given to the provisions of the *Prevention of Food Adulteration Act,* 1954.

0.5 The provisions on packing and marking (*see* **4**) are also subject to the requirements stipulated under the *Weights and Measures (Packaged Commodities) Rules,* 1977.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for drinking chocolate intended for use as a beverage.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of a powder or granules, free from lumps, having the colour, taste and flavour characteristic of the product. The product is essentially prepared out of cocoa powder and sugars. It may also contain food additives such as emulsifiers, antioxidant and flavouring agents which are permitted under the PFA Rules.

2.1.1 The product shall be free from rancidity or other off odours, insect infestation, fungus growth, added colouring matter, preservatives, adulterants and other harmful ingredients.

2.1.2 The material shall disperse satisfactorily without undue quick sedimentation and shall yield a product of good palatable and characteristic flavour and body.

2.1.3 The material shall be free from any other bat except cocoa butter.

2.2 As there is no internationally accepted method for the determination of dry fat free cocoa solids in drinking chocolate, the manufacturer is required to maintain an account of the quantity of cocoa powder added to each batch.

2.3 The material shall also comply with the requirements given in Table 1.

2.4 Raw Materials — The sugar and cocoa powder used in the manufacture of the product should conform to IS : 498-1970* and IS : 1164-1980† respectively.

3. PACKING AND MARKING

3.1 Packing — Unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in clean and sound tin or other airtight containers.

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*Grading for vacuum pan sugar (plantation white) (*third revision*).

†Specification for cocoa powder (*second revision*).

TABLE 1 REQUIREMENTS FOR DRINKING CHOCOLATE

(*Clauses* 2.3)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO		
			Appendix/Clause of This Standard	Appendix of IS : 1164-1969*	Related Indian Standard
(1)	(2)	(3)	(4)	(5)	(6)
i)	Moisture, percent by mass, *Max*	3	—	B	—
ii)	Total sugar (sucrose and/or dextrose), percent by mass, *Max*	75	A	—	—
iii)	Cocoa powder (expressed on as-is basis), percent by mass, *Min*	25	2.2	—	—
iv)	Acid-insoluble ash (on moisture-, fat- and sugar-free basis), percent by mass, *Max*	0·2	B	—	—
v)	*Staphylococcus aureus*	Absent	—	—	IS : 5887 (Part 2)-1976†
vi)	*Salmonella*	Absent	—	—	IS : 5887 (Part 3)-1976‡
vii)	*E. Coli* per 10 gm	Absent	—	—	IS : 5887 (Part 4)-1976§
viii)	Yeast and mould count, per gm, *Max*	100	—	—	IS : 5403-1969‖

*Specification for cocoa powders.

†Isolation, identification and enumeration of staphylococcus aureus faecal streptococci (*first revision*).

‡Isolation and identification of salmonella and shigella (*first revision*).

§Isolation, identification and enumeration of escherichia coli (*first revision*).

‖Method for yeast and mould count of foodstuffs.

3.2 Marking — The following particulars shall be marked or labelled legibly and indelibly on each container:

a) Name of the material;
b) List of ingredients;
c) Name of the manufacturer;
d) Batch or code number;
e) Net mass of contents; and
f) Date of manufacture.

3.2.1 Each container may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Markon products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions, under which a licence for the use of the Standard Mark may be granted to manufacturers or processors, may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 The method of drawing representative sample of the material and the criteria for conformity shall be as prescribed in Appendix C.

5. TESTS

5.1 Tests shall be carried out as prescribed in col 4 to 6 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

APPENDIX A

[*Table* 1, *Item* (ii)]

DETERMINATION OF TOTAL SUGAR (SUCROSE AND/OR DEXTROSE)

A-0. GENERAL

A-0.1 Before determining the sucrose content in the product, it is essential to find out whether dextrose is present or not. This method consists of a qualitative test (*see* **A-1**) which shall be performed to determine the presence of reducing sugars. If reducing sugars are present, the procedure given in **A-2** shall be adopted to identify if the reducing sugar present is dextrose.

A-0.2 If dextrose is absent, the sucrose content is estimated directly as given in **A-3**. In case dextrose is found to be present, it shall be estimated as given in **A-4** before estimating sucrose.

A-0.3 The total sugar shall be calculated finally as given in **A-5**.

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*Specification for water for general laboratory use (*second revision*).

A-1. QUALITATIVE TEST FOR PRESENCE OF REDUCING SUGARS

A-1.1 Procedure — Take approximately 10 g of the sample and add to it 100 ml distilled water. Boil the solution for a few minutes, clarify by adding 2 ml lead subacetate solution and filter. Test the filtrate with the Fehling's solution. A red precipitate of cuprous oxide indicates presence of reducing sugars. If red precipitate is present, proceed as given in **A-2** otherwise proceed as in **A-3**.

A-2. TEST TO CONFIRM THAT REDUCING SUGAR IS DEXTROSE

A-2.1 Reagents

A-2.1.1 *Ethyl Acetate*

A-2.1.2 *Glacial Acetic Acid* — analytical reagent grade.

A-2.1.3 p-*Anisidine*

A-2.1.4 *Phthalic Acid*

A-2.1.5 *Diphenylamine* — analytical reagent grade.

A-2.1.6 o-*Phosphoric Acid* — analytical reagent grade.

A-2.1.7 *Aniline* — analytical reagent grade.

A-2.1.8 *Acetone*

A-2.1.9 *Alcohol* — 95 percent.

A-2.1.10 *Lactose, Maltose, Glucose, Fructose*

A-2.1.11 *Anthrone*

A-2.1.12 *Solvents* — Mix ethyl acetate (for chromatography), glacial acetic acid and water in the proportion of 6 : 3 : 2. The resulting solvent has one phase and should be used immediately.

A-2.1.13 *Paper* — A paper suitable for sugar chromatography.

A-2.1.14 *Developers*:

a) *For reducing sugars* — Dissolve 1·23 g *p*-anisidine and 1·66 g phthalic acid in 100 ml acetone. Develop the dried chromatograms by dipping them into the solution and then drying in the oven at 102 to 105°C for 10 minutes. Reducing sugars show up as brown spots.

b) *For reducing and non-reducing sugars* — Mix 5 volumes of 4 percent alcoholic aniline solution with 5 volumes of 4 percent alcoholic diphenylamine solution and 1 volume of *o*-phosphoric acid.

Develop the dried chromatograms by spraying with this solution and drying in the oven at 100°C for 5 minutes. If the chromatogram is overheated, the whole paper becomes dark blue. The developed chromatograms only keep for a few hours.

c) *For fructose and fructose-containing saccharides* — Dissolve 0·4 g anthrone in 100 ml acetone and add 7 ml *o*-phosphoric acid. Dip the chromatograms into the solution and dry in the oven at 105°C for 5 minutes. Fructose and fructose-containing saccharides show up as spots which vary in colour from yellow to yellow-brown. Since these are insoluble in water, the excess phosphoric acid may be removed by rinsing the paper in water for a short while. On subsequent drying the chromatograms last very well.

A-2.1.15 *Marker Solution* — Boil the water containing a few grains of mercuric iodide for a few minutes. Cool the water and then use it to make solutions containing 0·3 percent of lactose, maltose glucose and fructose respectively. The solution lasts for months.

A-2.2 Procedure — To 5 g of finely grated chocolate placed in a centrifugal tube, add 10 ml of water. Place the tube in water-bath maintained at 50 to 60°C for 10 minutes and shake the centrifugal tube from time to time. Centrifuge the solution for 10 minutes at 3 000 to 3 500 rev/min. After this, place the tube into very cold water or in a refrigerator in order to solidify the fat which has separated as a layer on top. Pierce the fatty layer by a glass rod and filter the solution below through a small dry, fluted paper of a hardened variety. Collect about 0·5 ml of the pale brown and clear filterate. Apply about 10 ml of the filterate to a strip of chromatography paper by means of a platinum loop or, preferably, by means of a capillary pipette. In order to keep the spot as small as possible, the application may be done in two lots of 5 μl. Apply 5 μl of the marker solution at a distance of about 3 cm. Place the paper in the cabinet, add the relevant solution and run as a descending chromatogram for 14 to 16 hours. Then dry the strip by hanging it in air or in an oven at 30 to 40°C. It is now developed. If a particularly clear separation of lactose and maltose is required, the dried strip may be run with the same solvent for another night before being re-dried and developed.

A-2.2.1 The various sugars will give spots of different colouration which may also be examined under U. V. light:

Sugar	*Colour*
Maltose	Navy blue
Lactose	Blue-green
Sucrose	Green-yellow
Fructose	Yellow
Glucose	Browny-blue-green

A-3. DETERMINATION OF SUCROSE

A-3.1 Apparatus

A-3.1.1 *Glass Beaker* — 100 ml capacity.

A-3.1.2 *Volumetric Flasks* — 250 ml and 50 ml capacity.

A-3.1.3 *Filter Funnel* — 8 to 10 cm diameter and provided with pleated filter paper of diameter 15 cm.

A-3.1.4 *Pipette* — 50 ml, 5 ml.

A-3.1.5 *Conical Flask* — 50 ml capacity.

A-3.1.6 *Polarimeter*

A-3.2 Reagents

A-3.2.1 *Lead Subacetate (40 Percent)* — Dissolve 230 g of normal lead acetate [Pb (H_3CO_2)$_2$ $3H_2O$] in about 700 ml of boiling distilled water. While boiling continues, add gradually stirring 120 g of lead oxide (PbO). When the lead oxide has dissolved to the maximum possible, filter the solution into a litre flask. While it is hot, wash with boiling water, cool and make up to one litre. Mix well.

A-3.2.2 *Glacial Acetic Acid*

A-3.2.3 *Hydrochloric Acid* — 6·3 N.

A-3.2.4 *Potassium Oxalate Crystals*

A-3.3 Procedure

A-3.3.1 *Preparation of the Sample*

A-3.3.1.1 Weigh 25 g of the grated chocolate sample in a 100-ml beaker. Warm the sample until it becomes just fluid and stir to a cream with about 40-ml of distilled water at 80°C. Dilute with about 100 ml of warm distilled water and transfer the mixture to a 250-ml volumetric flask. Cool the flask and the contents with running water. Add 3·75 ml of lead subacetate, or 5 ml, if the fat-free cocoa solids are known to exceed 20 percent. Make up the volume at 20°C with distilled water and mix well.

A-3.3.1.2 Filter the mixture through a 15 cm No. 1 Whatman Pleated filter paper. Reject the first 20 ml of filtrate and collect the remainder.

A-3.3.1.3 Measure 50 ml of the filtrate into a dry flask or stoppered cylinder, add 0·2 ml of glacial acetic acid and mix well.

A-3.3.2 *Readings of Zero Error of Polarimeter* — Take a series of polarimeter readings of distilled water in a standard 2-dm polarimeter tube. Take care that the temperature is exactly 20°C and that the signs (+) or (—) are properly set down. Divide the algebraic sum of the readings by the number of readings. The value so obtained gives the zero error, if any, of the polarimeter. For this and other readings, if a jacketed tube is available, the liquid in the tube may be cooled or warmed to the correct temperature by water circulated through the jacket. A more convenient method is to take readings in a room maintained at a constant temperature of 20°C. When a jacket tube is used, it is preferably filled through the aperture in which the thermometer is to be inserted. Small errors are caused through strains set up in the cover glasses by unscrewing the ends of the tube. But this is unavoidable when filling a tube without a jacket. Rinse the tube several times with the solution to be tested prior to filling for taking readings.

A-3.3.3 *Readings of Test Solution (Polarimeter Readings Before Inversion)* — Record a series of readings at 20°C of the prepared solution (**A-3.3.1.3**) in the same tune as was used for the readings of zero error, paying careful attention to the sign. Deduct the zoro error algebraically. Allow for the dilution due to acetic acid by multiplying the difference by 1·004.

A-3.3.4 *Procedure for Inversion (Polarimeter Readings after Inversion)* — Pipette 40 ml of the clarified filtrate (**A-3.3.1.2**) from the 10 percent solution into a 50 ml volumetric flask· Add 5 ml of 6·3 N hydrochloric acid solution and insert a thermometer. Place the flask in a water-bath containing sufficient water at about 62°C to bring the temperature of the contents of the flask to 60°C in 2 minutes, whilst swirling the flask, immersed so that the level of the contents is well below the level of the water in the bath. Maintain the temperature of the solution at 60°C for 10 minutes. Remove the flask from the bath, cool it immediately, remove the thermometer from the flask, rinse it and make up to 50 ml at 20°C and mix. Take the polarimeter reading after one hour. Deduct the zero reading and multiply by 1·25 to allow for dilution.

A-3.4 Calculation of Sucrose

A-3.4.1 Specific rotation (D) of the sample before inversion is calculated as follows:

$$D = \frac{100 \times d}{L \times C}$$

where

d = the direct angular rotation, corrected (*see* **A-3.3.3**);

L = length of tube in decimetres; and

C = the concentration of the solution in respect to the sample (10 g per ml in this case).

A-3.4.2 Specific rotation of the sample after inversion is cacleulated as follows:

$$I = \frac{100.d}{L.C}$$

where

d = the direct angular rotation, corrected (*see* **A-3.3.4**);
L = length of tube in decimetres; and
C = the concentration of the solution in respect to the sample (10 g per ml in this case).

A-3.4.3 The percentage of sucrose (S) is calculated as follows:

$$S = \frac{D - I}{0{\cdot}881\ 1}$$

where

D = specific rotation of the sample before inversion (**A-3.4.1**),
I = specific rotation of the sample after inversion (**A-3.4.2**), and
0·881 1 = conversion factor for sucrose.

A-3.4.3.1 This sucrose content 'S' should be multiplied by a correction factor 'V' for the volume occupied by the lead precipitate and other insoluble matter. For a 10 percent solution, the correction factor shall be calculated:

$$V = \frac{1\ 000 - (\ 1{\cdot}02\ F + 0{\cdot}8\ P\ )}{1\ 000}$$

where

F = the percentage of fat, and
P = the percentage of fat-free dry cocoa plus milk protein in the sample.

A-3.4.3.2 'F' may be found by analysis (*see* Appendix B of IS : 1163-1971*) calculated sufficiently closely by deducting the sum of the percentages of moisture + fat + uncorrected sucrose + uncorrected lactose (if present) from 102·5. The figure 102·5 allows for sugars (sucrose and lactose) content of approximately 50 percent. In most cases the correction factor 'V' will be approximately 0·98.

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*Specification for chocolate (*first revision*).

A-4. DETERMINATION OF DEXTROSE

A-4.1 Reagents

A-4.1.1 *Stock Solution of Dextrose* — Accurately weigh 10 g of anhydrous dextrose into a one-litre graduated flask and dissolve it in water. Add to this solution 2·5 g of benzoic acid, shake to dissolve the benzoic acid and make up the volume to the mark with water. (This solution should not be used after 48 hours).

A-4.1.2 *Standard Dextrose Solution* — Dilute a known aliquot of the stock solution of dextrose (*see* **A-4.1.1**) with water containing 0·25 percent (*m/v*) of benzoic acid to such a concentration that more than 15 ml but less than 50 ml of it will be required to reduce all the copper in the Fehling's soultion taken for titration. Note the concentration of anhydrous dextrose in this solution as milligrams per 100 ml (*see* Note 1). Prepare this solution fresh every day.

NOTE 1 — When 10 ml (*see also* **A-4.3.1.1**) of Fehling's solution are taken for titration, a standard dextrose solution containing 0·11 to 0·30 percent (*m/v*) of anhydrous dextrose is convenient for use.

A-4.1.3 *Methylene Blue Indicator Solution* — Dissolve 0·2 g of methylene blue in water and dilute to 100 ml.

A-4.1.4 *Petroleum Ether* — redistilled below 60°C.

A-4.1.5 *Fehling's Solution* (*Soxhlet Modification*) — Prepared by mixing immediately before use, equal volumes of solution A and solution B.

A-4.1.5.1 *Solution A* — Dissolve 34·639 g of copper sulphate ($CuSO_4$, $5H_2O$) in water, add 0·5 ml of concentrated sulphuric acid of sp gr 1·84 (conforming to analytical reagent grade of IS : 266-1950*), and dilute to 500 ml in a graduated flask. Filter the solution through prepared asbestos.

A-4.1.5.2 *Solution B* — Dissolve 173 g of Rochelle salt [potassium sodium tartrate ($KNaC_4H_4O_6$, $4H_2O$)] and 50 g of sodium hydroxide analytical reagent (conforming to IS : 376-1976†) in water, dilute to 500 ml in a graduated flask. Filter the solution through prepared asbestos.

A-4.1.6 *Standardization of Fehling's Solution* — Pour standard dextrose solution (*see* **A-4.1.2**) into a 50 ml burette (*see* Note 6). Find the titre (that is the volume of standard dextrose solution required to reduce all the copper in 10 ml of Fehling's solution) corresponding to the concentration of standard dextrose solution from Table 2. (If, for example, the

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*Specification for sulphuric acid (*revised*).

†Sodium hydroxide, analytical grade (*second revision*).

standard dextrose solution contains 167·0 mg of anhydrous dextrose per 100 ml, the corresponding titre should be 30 ml). Pipette 10 ml (*see also* **A-4.3.1.1**) of Fehling's solution into a 300 ml conical flask and run in from the burette almost the whole of the standard dextrose solution required to effect reduction of all the copper, so that not more than one millilitre will be required later to complete the titration. Heat the flask containing the mixture over a wire gauze. Gently boil the contents of the flask for 2 min. At the end of 2 min of boiling, add without interrupting boiling, one millilitre of methylene blue indicator solution. While the contents of the flask continue to boil, begin to add standard dextrose solution (one or two drops at a time) from the burette till the blue colour of the indicator just disappears.

The titration should be completed within one minute, so that the contents of the flask boil altogether for 3 min without interruption (*see* Note 5). Note the titre (that is the total volume in millilitres of standard dextrose solution used for the reduction of all the copper in 10 ml of Fehling's solution). Multiply the titre (obtained by direct titration) by the number of milligrams of anhydrous dextrose in one millilitre of the standard dextrose solution to obtain the dextrose factor. Compare this factor with the dextrose factor given in Table 1 and determine the correction, if any, to be applied to the dextrose factor derived from Table 2.

Example

Concentration in mg/100 ml of anhydrous dextrose in standard dextrose solution = 167·0

Titre in ml obtained by direct titration = 30·1

Dextrose factor for 30·1 ml of standard dextrose solution = titre in ml × number of mg of anhydrous dextrose in one millimetre of standard dextrose solution

= 30·1 × 1·670

= 50·2670

Dextrose factor for 30·1 ml of standard dextrose solution from Table 1 (calculated by interpolation) = 50·11

Correction to be applied to the dextrose factor derived from Table 1 = 50·267 0 — 50·110 0

= + 0·157 0

A-4.2 Procedure

A-4.2.1 *Preparation of the Sample* — Proceed as for sucrose (*see* **A-3.3.1**) till step **A-3.3.1.2.** Take 100 ml of this filtrate in a beaker, add 0·5 g of potassium oxalate. Mix and allow to stand for 15 to 20 minutes, mixing occasionally and then filter.

A-4.2.2 *Incremental Method of Titration* — Pour the prepared solution (*see* **A-4.2.1**) into a 50 ml burette (*see* Note 6). Pipette 10 ml of Fehling's solution into a 300 ml conical flask and run in from the burette 15 ml of the prepared solution. Without further dilution, heat the contents of the flask over a wire gauze, and boil. (After the liquid has been boiling for about 15 s, it will be possible to judge if almost all the copper is reduced by the bright red colour parted to the boiling liquid by the suspended cuprous oxide). When it is judged that nearly all the copper is reduced, add one millilitre of methylene blue indicator solution (*see* Note 2). Continue boiling the contents of the flask for one to two minutes from the commencement of ebullition, and then add the prepared solution in small quantities (one millilitre or less at a time), allowing the liquid to boil for about 10 s between successive additions till the blue colour of the indicator just disappears (*see* Note 5). In case there still appears to be much unreduced copper after the mixture of Fehling's solution with 15 ml of the prepared solution has been boiling for 15 s, add the prepared solution from the burette in large increments (more than one millilitre at a time according to judgement), and allow the mixture to boil for 15 s after each addition. Repeat the addition of the prepared solution at intervals of 15 s until it is considered unsafe to add a large increment of the prepared solution. At this stage, continue the boiling for an additional one to two minutes, add one millilitre of methylene blue indicator solution and complete the titration by adding the prepared solution in small quantities (less than one millilitre at a time (*see also* Note 3).

NOTE 2 — It is advisable not to add the indicator until the end point has been nearly reached because the indicator retains its full colour until the end point is almost reached and thus gives no warning to the operator to go slowly.

NOTE 3 — When the operator has had a fair amount of experience with the method, a sufficiently accurate result may often be obtained by a single estimation by the incremental method of titration. For the utmost degree of accuracy of which the method is cabable, a second titration should be carried out by the standard method of titration (*see* **A-4.2.3**).

A-4.2.3 *Standard Method of Titration* — Pipette 10 ml of Fehling's solution into a 300 ml conical flask and run in from the burette almost the whole of the prepared solution required to effect reduction of all the copper (*see* **A-4.2.2**), so that, if possible, not more than one millilitre will be required later to complete the titration. Gently boil the contents of the flask for 2 min. At the end of 2 min of boiling, add without interrupting boiling, one millilitre of methylene blue indicator solution. While the contents of the flask continue to boil, begin to add the prepared solution (one or two drops at a time) from the burette till the blue colour of the indicator just disappears (*see* Note 4). [The titration should be completed within one minute so that the contents of flask boil altogether for 3 min without interruption (*see* Note 5).]

In case of doubt, the flame may be removed from the wire gauze for one or two seconds and the flask held against a sheet of white paper. (A holder of paper, suitably fixed round the neck of the flask, is very convenient for this purpose as it can be left round the neck of the flask without risk of over balancing it). The top edge of the liquid would appear bluish if the indicator is not completely decolourized. It is inadvisable to interrupt the boiling for more than a few seconds as the indicator undergoes back oxidation rather rapidly when air is allowed free access into the flask, but there is no danger of this as long as a continuous stream of steam is issuing from the mouth of the flask.

NOTE 4 — The indicator is so sensitive that it is possible to determine the end point withing one drop of the prepared solution in many cases. The complete decolouration of the methylene blue is usually indicated by the whole reaction liquid, in which the cuprous oxide is continuously churned up, becoming bright red or orange in colour.

NOTE 5 — It should be observed that with both incremental and standard methods of titration, the flask containing the reaction mixture is left on the wire gauze over the flame throughout the titration, except when it may be removed for a few seconds to ascertain if the end point is reached.

NOTE 6 — In adding sugar solution to the reaction mixture, the burette may be held in hand over the flask. The burette may be fitted with a small outlet tube bent twice at right angles, so that the body of the burette can be kept out of the steam while adding sugar solution. Burettes with glass taps are unsuitable for this work, as the taps become beated by the steam and liable to jam.

A-4.3 Calculation

A-4.3.1 Refer to Table 2 for the dextrose factor corresponding to the titre and apply the correction previously determined under. Calculate the percentage of dextrose in the sample as follows:

$$\text{Percent dextrose, } D = \frac{M}{X} \times 0{\cdot}98$$

where

M = mass of dextrose in milligrams read from the table after applying the correction;

X = titre value; and

0·98 = correction factor for volume occupied by insoluble matter.

A-4.3.1.1 Instead of using 10 ml of Fehling's solution, a 25 ml portion may also be substituted throughout the procedure (including 'standardization of Fehling's solution' under **A-4.1.6**). In this case, the standard dextrose solution, used in standardizing the Fehling's solution and the prepared solution of the material (*see* **A-4.2.1**) shall contain 0·25 to 0·75 percent (*m*/*v*) of anhydrous dextrose, and Table 3 shall be used for all calculation.

A-5. CALCULATION OF TOTAL SUGAR

A-5.1 The total sugar, percent $= S + D$

where

S = percent sucrose (*see* **A-3.4**), and

D = percent dextrose (*see* **A-4.3**).

APPENDIX B

[*Table* 1, *Item* (iv)]

DETERMINATION OF ACID INSOLUBLE ASH

B-1. APPARATUS

B-1.1 Furnace — going up to at least 650°C.

B-1.2 Silica Dish

B-2. REAGENTS

B-2.1 Petroleum Ether — Redistilled boiling range 40 to 60°C.

B-2.2 Dilute Hydrochloric Acid — Approximately 5 N, prepared from concentrated hydrochloric acid.

B-3. PROCEDURE

B-3.1 Weigh accurately about 10 g of the material in a silica dish, of known mass. Heat the material in the dish at 100°C until water is expelled and then heat slowly over a flame until swelling ceases. Complete the ignition by keeping in a muffle furnace at 550°C. Continue the ashing until there is no further change in the colour of the inorganic residue, that is, until the latter is present as a white powder. Normally this takes three hours. If it is not white till then, moisten the ash with a few drops of petroleum ether and, evaporate it by warming the dish. Ash the sample for another thirty minutes at 550°C to 600°C. This treatment shall be continued until a white residue is formed. Cool the dish and weigh.

B-3.2 To the ash contained in the dish, add 25 ml of dilute hydrochloric acid, cover the dish with a watch glass and heat it on a boiling water-bath for 10 minutes. Allow to cool and filter the contents of the dish through Whatman filter paper No. 42 or its equivalent. Wash the filter paper till the washings are free from the acid. Return the filter paper and the residue to the dish. Keep it in an electric air oven maintained at 105 ± 2°C for about 3 hours. Ignite in a muffle furnace at 550°C for one hour. Cool the dish in a desiccator and weigh. Repeat the process of igniting in a muffle furnace, cooling and weighing at half hour intervals until the difference in mass between the successive weighings is less than 1 mg. Note the lowest mass.

B-4. CALCULATION

B-4.1 Acid — Insoluble ash on moisture-, fat- and sugar-free basis (A) shall be calculated as follows:

$$A = \frac{10\,000\, m}{M\, 100 - (F + M + S)}$$

where

m = mass in g of acid-insoluble ash,
M = mass in g of the sample taken for test,
F = percent fat content of sample,
M = percent moisture in sample, and
S = percent sugar in sample.

APPENDIX C

(*Clause* 4.1)

SAMPLING OF DRINKING CHOCOLATE

C-1. GENERAL REQUIREMENTS

C-1.0 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

C-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

C-1.2 The sampling instrument, preferably a spoon or spatula, shall be clean and dry when used.

C-1.3 The samples, the material being sampled, the sampling instrument and the containers for samples, shall be protected from adventitious contamination.

C-1.4 The samples shall be placed in clean and dry glass or tin cantainers. The sample containers shall be of such a size that they are almost completely filled by the sample.

C-1.5 Each container shall be sealed air-tight after filling and marked with full details of sampling, batch or code number, name of the manufacturer, sub-group number and other important particulars of the consignment and lot.

C-1.6 Samples shall be stored in such a manner that they are protected from light and the temperature of the material does not vary unduly from the normal temperature.

C-2. SCALE OF SAMPLING

C-2.1 Lot — All the containers of the same size in a single consignment of material drawn from a single batch of manufacture shall constitute a lot.

C-2.2 Samples shall be tested for each lot separately for ascertaining conformity of the material to the requirements of this specification. The total number of containers to be selected from the lot shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Tables 4 and 5.

C-2.2.1 These containers shall be chosen at random from the lot and for this purpose a random number table (*see* IS : 4905-1962*) as agreed to between the purchaser and the vendor shall be used. In case such a table is not available, the following procedure shall be adopted:

> Starting from any container, count in one order as 1, 2, 3...... up to r and so on, where r is the integral part of N/n (N being the number of containers in the lot and n the number of containers to be selected). Every rth container so counted shall be withdrawn to constitute the sample.

C-3. TEST SAMPLES AND REFEREE SAMPLES

C-3.1 The sample containers of net contents less than 500 g selected according to **C-2.2** and col 1 and 2 of Table 4 shall be equally divided at random into a number of groups specified in col 3 of Table 4. Each sample container of net content 500 g or more selected according to **C-2.2** and col 1 and 2 of Table 5 shall be treated as one group.

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*Methods for random sampling.

C-3.2 Preparation of Individual Samples — The contents of all the containers in a group shall be poured out and mixed thoroughly. About 360 g of the material shall be taken from this and divided into three equal parts. Each part, so obtained, shall be transferred to sample containers which shall be sealed air-tight and labelled with the particulars given in **C-1.5**. The samples so obtained shall be divided into three sets in such a way that each set has a sample representing each group. One of these sets shall be marked for the purchaser, another for the vendor and the third for the referee.

C-3.3 Preparation of a Composite Sample — From the mixed material of each group remaining after taking the sample in **C-3.2**, equal quantities of the material shall be taken and mixed up together so as to form a composite sample representing the lot as a whole and weighing not less than 120 g. This composite sample shall be divided into three equal parts and transferred to clean, dry sample containers and labellel with all particulars given in **C-1.5**. One of these composite samples representing the lot as a whole shall be for the purchaser, another for the vendor and the third for the referee.

C-3.4 Referee Sample — Referee samples for a lot shall consist of a set of samples obtained in **C-3.2** and a composite sample obtained in **C-3.3** marked for this purpose and shall bear the seals of the purchaser and the vendor. These shall be kept at a place and under conditions agreed to between the purchaser and the vendor.

C-4. NUMBER OF TESTS

C-4.1 The tests for the description, cocoa powder and moisture shall be conducted on the individual samples as obtained in **C-3.2**.

C-4.2 The tests for sugar and crude fibre shall be made on the composite sample as prepared under **C-3.3**.

C-5. CRITERIA FOR CONFORMITY

C-5.1 The lot shall be considered as conforming to the requirements of this specification if **C-5.1.1** and **C-5.1.2** are satisfied.

C-5.1.1 The results of the tests conducted on the individual samples, for the characteristics specified in **C-4.1** shall satisfy the corresponding requirement as given in **3**.

C-5.1.2 The results of the tests conducted on composite sample for the various characteristics mentioned in **C-4.2** shall satisfy the corresponding requirements as given in **3**.

TABLE 2 DEXTROSE FACTORS FOR 10 ml OF FEHLING'S SOLUTION

(*Clause* A-4.1.6)

TITRE (1) ml	DEXTROSE FACTORS* (2)	DEXTROSE CONTENT, PER 100 ml OF SOLUTION (3) mg
15	49·1	327
16	49·2	307
17	49·3	289
18	49·3	274
19	49.4	260
20	49·5	247·4
21	49·5	235·8
22	49·6	225·5
23	49·7	216·1
24	49·8	207·4
25	49·8	199·3
26	49.9	191·8
27	49·9	184·9
28	50·0	178·5
29	50·0	172·5
30	50·1	167·0
31	50·2	161·8
32	50·2	156·9
33	50·3	152·4
34	50·3	148·0
35	50·4	143·9
36	50·4	140·0
37	50·5	136·4
38	50·5	132·9
39	50·6	129·6
40	50·6	126·5
41	50·7	123·6
42	50·7	120·8
43	50·8	118·1
44	50·8	115·5
45	50·9	113·0
46	50·9	110·6
47	51·0	108·4
48	51·0	106·2
49	51·0	104·1
50	51·1	102·2

*Milligrams of anhydrous dextrose corresponding to 10 ml of Fehling's solution.

TABLE 3 DEXTROSE FACTORS FOR 25 ml OF FEHLING'S SOLUTION

(*Clause* A-4.3.1.1)

TITRE	DEXTROSE FACTOR*	DEXTROSE CONTENT, PER 100 ml OF SOLUTION
(1)	(2)	(3)
ml		mg
15	120·2	801
16	120·2	751
17	120·2	707
18	120·2	668
19	120·3	638
20	120·3	601·5
21	120·3	572·9
22	120·4	547·3
23	120·4	523·6
24	120·5	501·9
25	120·5	482·0
26	120·6	463·7
27	120·6	446·8
28	120·7	431·1
29	120·7	416·4
30	120·8	402·7
31	120·8	389·7
32	120·8	377·6
33	120·9	366·3
34	120·9	355·6
35	121·0	345·6
36	121·0	336·3
37	121·1	327·4
38	121·2	318·8
39	121·2	310·7
40	121·2	303·1
41	121·3	295·9
42	121·4	289·0
43	121·4	282·4
44	121·5	276·1
45	121·5	270·1
46	121·6	264·3
47	121·6	258·8
48	121·7	253·5
49	121·7	248·4
50	121·8	243·6

NOTE — Tables 1 and 2 show, for the standard method of titration, the values corresponding to integral millilitres of the sugar solutions, intermediate values being obtained by interpolation.

*Milligrams of anhydrous dextrose corresponding to 25 ml of Fehling's solution.

TABLE 4 SAMPLING OF CONTAINERS OF NET CONTENT LESS THAN 500 g

(*Clauses* C-2.2 *and* C-3.1)

NUMBER OF CONTAINERS IN THE LOT (1) *N*	TOTAL NUMBER OF CONTAINERS TO BE SELECTED (2) *n*	NUMBER OF GROUPS INTO WHICH SAMPLE CONTAINERS HAVE TO BE DIVIDED (3)
Up to 50	9	1
51 to 300	18	2
301 to 500	20	2
501 to 1 000	30	3
1 001 to 3 000	40	4
3 001 and above	50	5

TABLE 5 SAMPLING OF CONTAINERS OF NET CONTENT 500 g OR MORE

(*Clauses* C-2.2 *and* C-3.1)

NUMBER OF CONTAINERS IN THE LOT (1) *n*	TOTAL NUMBER OF CONTAINERS TO BE SELECTED (2) *n*
Up to 50	2
51 to 300	3
301 to 500	4
501 to 1 000	5
1 000 and above	6

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IS : 6762 - 1986 SPECIFICATION FOR DRINKING CHOCOLATE (SWEETENED COCOA POWDER)

(*Second Revision*)

(*Page* 18, *clause* **C-2.2.1**, *line* 2) — Substitute '1968' *for* '1962'.

(*Page* 18, *foot-note marked with* '*' *mark*) — Substitute 'Methods for random sampling (*first revision*)' for the existing foot-note.

(*Page* 22, *Table* 5, *col* 1) — Substitute '*N*' *for* '*n*' and '1 001 and above' *for* '1 000 and above'.

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(Second Revision)

[*Page* 5, *clause* **3.2**, *Sl No.* (e)] — Substitute 'Net quantity' *for* 'Net mass'.

(*Page* 5, *clause* **3.2**) — Insert a new Sl No. (g) after Sl No. (f) as under:

'g) Any other requirements as given under the *Standards of Weights and Measures (Packaged Commodities) Rules*, 1977 and the *Prevention of Food Adulteration Act,* 1955 and the Rules.'

(FAD 6)

Reprography Unit, BIS, New Delhi, India